

Supplemental Paper S1: Analytical details

Whole-rock analyses

XRF at the University of Stellenbosch

Whole-rock major-element analyses were carried out by XRF, using glass disks made in an autofluxer, with lithium borate flux but without any heavy absorber. Internal standards were basalt BHVO-1 and granite NIM-G. For the granite, calculated relative uncertainties (twice the standard errors for the NIM-G, in wt%) are; SiO₂ = 0.363, TiO₂ = 0.006, Al₂O₃ = 0.516, FeO^T = 0.043, MnO = 0.001, MgO = 0.005, CaO = 0.016, Na₂O = 0.071, K₂O = 0.055 and P₂O₅ = 0.005. Results are reported normalized to 100 wt% anhydrous, with total Fe expressed as FeO^T.

LA-ICP-MS at Stellenbosch

Trace elements were analysed by Laser-ablation ICP-MS on the same glass disks, using a standard BHVO-1 glass made with the same flux as the samples. The standard was run at the beginning and the end of the sample string and after each 5 sample measurements. Drift corrections were then applied. All measurements were in duplicate, with the average reported as the measured concentration. Further queries regarding analytical methods should be addressed to the Central Analytical Facility (CAF) at Stellenbosch University. Table S1.1 shows the percentage relative deviation of the measured to the certified values for the standard.

Table S1.1 Percentage relative deviations of measured concentration from certified values in fluxed glass made from standard basalt BHVO-2

| Element | RSD % | Element | RSD % | Element | RSD % |
|---------|-------|---------|-------|---------|-------|
| Rb | 0.4 | Ce | 3.8 | Tm | 6.4 |
| Sr | 2.5 | Pr | 12.6 | Yb | 11.4 |
| Ba | 4.1 | Nd | 9.0 | Lu | 17.1 |
| Sc | 5.3 | Sm | 2.5 | Hf | 7.5 |
| Cr | 1.0 | Eu | 20.2 | Ta | 4.0 |
| Ni | 7.6 | Gd | 4.0 | Pb | 6.2 |
| Y | 4.1 | Tb | 2.2 | Th | 10.7 |
| Zr | 5.2 | Dy | 9.0 | U | 6.3 |
| Nb | 5.8 | Ho | 9.3 | | |
| La | 9.6 | Er | 6.4 | | |

Sr and Nd Isotopes by solution MC-ICP-MS at the University of Cape Town

For each sample, approximately 50 mg of powder was weighed into a Teflon beaker and about 4 ml 4:1 concentrated 2B HF:HNO₃ was added. The beaker was then capped and left to digest on a hot plate for at least 2 days. The resulting solution was dried and re-dissolved in 6M 2B HNO₃, twice. After the second drying, the sample was taken up in 1.5 ml of 2M 2B HNO₃. After centrifuging, the supernatant was loaded onto a cleaned and pre-conditioned column of 0.2 mL Sr.Spec resin (Eichrom). The column was washed with 2M 2B HNO₃ and the Sr fraction collected in 0.02M 2B HNO₃ (after Míková and Denková, 2007; Pin and Zalduegui, 1997; Pin et al., 1994). Following the final drying, these fractions were re-dissolved in 2 ml of 0.2% HNO₃. Based on the XRF values for Sr concentrations of the original samples, 3 ml of 200 ppb Sr solutions were prepared for analysis using the same 0.2% HNO₃.

Analyses were performed using a NuPlasma HR MC-ICP-MS (Nu Instruments, Wrexham, Wales, UK) in the Department of Geological Sciences at the University of Cape Town. The approximately 200 ppb Sr sample and standard solutions were aspirated into the plasma through

a microcyclonic spraychamber. The on-peak background was measured for 120 seconds while aspirating the same 0.2% HNO₃ used to dilute the sample and standard solutions. These background measurements, including any krypton (⁸⁴Kr and ⁸⁶Kr) present in the argon gas, were subtracted from the measured signals. Instrumental mass fractionation was corrected using the exponential law and a fractionation factor based on the measured ⁸⁶Sr/⁸⁸Sr ratio and the accepted value of 0.1194. The ⁸⁷Rb contribution to the 87 amu signal was calculated and subtracted using this fractionation factor, the exponential law, the measured ⁸⁵Rb signal and a ⁸⁵Rb/⁸⁷Rb ratio of 0.3856.

To assess instrument tuning and stability, a 200 ppb Sr solution of the NIST SRM987 international Sr isotope standard was analysed twice, prior to any samples. The external, measured 2σ reproducibility of SRM987 was 0.000019 (n = 3) on an average ⁸⁷Sr/⁸⁶Sr ratio of 0.710277. All ⁸⁷Sr/⁸⁶Sr data were normalised to 0.710255, the in-house long-term average, which agrees with published results. The average ⁸⁴Sr/⁸⁶Sr ratio was 0.05643 ± 0.00012 (n = 4), also in agreement with published values.

Nd isotope analyses were carried out using the same multi-collector ICP-MS instrument that was used for the Sr isotope determinations, using the same primary solutions that were used for the Sr isotope work. Nd isotopes were analysed in 1.5 ml of 50 ppb Nd 2% HNO₃ solutions using a Nu Instruments DSN-100 desolvating nebuliser. JNdi-1 was used as bracketing standard, and all Nd isotope data presented are referenced to this standard, using a ¹⁴⁴Nd/¹⁴³Nd ratio of 0.512115 (Tanaka et al., 2000). All Nd isotope data were corrected for Sm and Ce interferences and for instrumental mass fractionation, using the exponential law and a ¹⁴⁶Nd/¹⁴⁴Nd value of 0.7219. For further details of the analytical techniques see Will et al. (2007). Analyses of Rb, Sr, Sma and Nd that were use to apply age corrections to the isotope ratios were carried out, by quadrupole ICP-MS, at UCT, on the same solutions that were used for the measurement of the Sr and Nd isotope ratios.

Zircon U-Pb dating by SC-SF-ICP-MS at Stellenbosch University Central Analytical Facilities

Zircons were isolated from the samples by mineral separation techniques, making use of standard crushing, milling, panning, Frantz magnetic separation and dense liquid separation. Zircon samples were mounted in 25 mm epoxy resin discs and polished to expose the interiors of the grains. All zircon crystals were then imaged using a Zeiss Merlin scanning electron microscope equipped with a CL detector.

Laser-ablation U-Pb isotope data were collected using a 193 nm wavelength ASI Resolution laser-ablation system coupled to a Thermo Scientific Element 2 single-collector magnetic sector field, inductively coupled plasma mass spectrometer (SC-SF-ICP-MS). U-Pb data and metadata are tabulated in Supplementary Tables 1 and 2. The interpretation of the U-Pb data here reported was carried out solely by the authors of this paper.

At the start of each analytical session the mass spectrometer was tuned by ablating a line scan on the NIST610 glass. The torch position, lenses and gas flows were tuned while measuring ²⁰⁶Pb, ²³⁸U and ²³⁸U¹⁶O, to obtain stable signals and high intensities on ²⁰⁶Pb and ²³⁸U, as well as low oxide rates (²³⁸U¹⁶O/²³⁸U < 0.3%). The ablated zircon material was transported from the laser cell into the ICP-MS using a continuous flow of 0.35 l/min of He gas. Signals of m/z 202, 204, 206, 207, 208, 232, 235 and 238 were acquired in electrostatic scan mode. The laser and ICP-MS software packages were synchronized to allow automated execution of the analysis. Each analytical session included up to 500 measurements.

The laser-sampling protocol employed a 25 μm static spot and a fluence of 2.0 J/cm². Before the gas blank measurement, each spot was pre-ablated by firing two laser shots. This served to remove common Pb from the surface, which could have been introduced during sample preparation.

During each analytical session, the zircon reference materials GJ-1 (Jackson et al., 2004), Plešovice (Sláma et al., 2008) and 91500 (Wiedenbeck et al., 1995) were measured between groups of 12 unknowns. Zircon GJ-1 was used as a matrix-matched primary reference material, to correct for mass discrimination on measured isotope ratios in unknown samples, with simultaneous correction for instrumental drift. The values used for normalisation are based on ratios determined by ID-TIMS reported in Horstwood et al. (2016). Plešovice and 91500 were used as secondary reference materials to validate the results and to assess the data quality in each analytical session.

Data reduction was performed with the software package Iolite v.3.5 (Paton et al., 2011), combined with VizualAge (Petrus and Kamber 2012). An exponential model of laser-induced elemental fractionation (LIEF), obtained by combining the isotope ratios of the primary reference material from the entire session, is used to correct for time-dependent, down-hole elemental fractionation in the unknowns, using the assumption that the same fractionation occurred in both the reference material and the unknowns. After correction for LIEF, and drift and normalisation to the main reference material (performed in Iolite), uncertainty components for systematic errors are propagated by quadratic addition, according to the recommendations of Horstwood et al. (2016). Table S1.2 provides the detailed metadata for this analytical protocol.

Table S1.2 U-(Th)-Pb dating method, CAF, Stellenbosch University, South Africa

| Laboratory & Sample Preparation | |
|--|---|
| Laboratory name | Central Analytical Facilities, Stellenbosch University, South Africa |
| Sample type / mineral | Igneous / zircons |
| Sample preparation | Conventional mineral separation, 1 inch resin mount, 1 μm polish to finish |
| Imaging | CL, ZEISS Merlin SEM, 11 nA, 10kV, 10 mm working distance |
| Laser ablation system | |
| Make, Model & type | ASI Resolution SE Excimer laser |
| Ablation cell & volume | Laurin Technic S-155 dual-volume cell |
| Laser wavelength | 193 nm |
| Pulse width | 5 ns SE |
| Fluence | 2.0 J/cm ² |
| Repetition rate | 7 Hz |
| Spot size | 25 μm |
| Sampling mode / pattern | Static spot ablation |
| Carrier gas | 100% He in the cell, Ar make-up gas combined using a T-connector close to sample cell |
| Pre-ablation surface cleaning | 2 pulses, same spot size as during ablation |
| Ablation duration | 29 s |
| Wash-out delay | 20 s |
| Cell carrier gas flow | 0.35 l/min He |
| ICP-MS Instrument | |
| Make, Model & type | Thermo Fisher Scientific Element2 single collector HR-SF-ICP-MS |
| Sample introduction | Ablation aerosol via conventional tubing |
| RF power | 1325 W |
| Make-up gas flow | 0.9 l/min Ar |
| Detection system | Single-collector Secondary Electron Multiplier |
| Masses measured | 202, 204, 206, 207, 208, 232, 235, 238 |
| Integration time per peak (segment duration) | 7, 14, 15, 18, 8, 8, 1, 13 ms, respectively |

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|--|---|
| Total integration time per output data point | 0.1 s |
| Sensitivity | 0.3% U |
| Dead time | 6 ns |
| Data Processing | |
| Gas blank | 13 s on peak prior to each measurement |
| Calibration strategy | GJ-1 used as primary reference material, Plešovice and 91500 used as secondary reference materials |
| Reference Material info | GJ-1 (Jackson et al., 2004; revised isotopic ratios from Horstwood et al., 2016); Plešovice (Sláma et al., 2008); 91500 (Wiedenbeck et al., 1995) |
| Data processing package used / Correction for LIEF | Iolite reduction software package v.3.5 with VizualAge; LIEF modelled within each analytical session on the basis of combined analyses of the main reference material; LIEF correction assumes reference material and samples behave identically. All ages and uncertainties calculated with Isoplot v. 4.15 (Ludwig, 2012) |
| Mass discrimination | Reference material-sample bracketing with $^{207}\text{Pb}/^{206}\text{Pb}$ and $^{206}\text{Pb}/^{238}\text{U}$ normalized to zircon GJ-1 |
| Common-Pb correction | No common Pb correction applied to the data |
| Uncertainty level & propagation | Decay constant uncertainties, ratio uncertainty of primary reference material and long-term excess variance of secondary reference material are propagated by quadratic addition. Age uncertainties are quoted at the 2σ absolute level. |
| Quality control / Validation | Plešovice: wtd ave $^{206}\text{Pb}/^{238}\text{U}$ age = 341.1 ± 1.1 Ma (95% conf, MSWD = 1.1; n = 46); 91500: wtd ave $^{206}\text{Pb}/^{238}\text{U}$ age = 1064.3 ± 4.1 Ma (95% conf, MSWD = 0.7; n = 27) |
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